

L# ANSWER 13 OF 419 CAPLUS COPYRIGHT 2002 ACS

AN 2001:927333 CAPLUS

DN 136:53678

TI N-methyl-2-pyrrolidone composition and method for its preparation

IN Ise, Yoko; Takahashi, Kazushige

PA Mitsubishi Chemical Corp., Japan

SO Jpn. Kokai Tokkyo Koho, 7 pp.

CODEN: JKXXAF

PATENT NO.      KIND      DATE      APPLICATION NO.      DATE

PI JP 2001354647      A2 20011225      JP 2000-177704      20000614

AB A N-methyl-2-pyrrolidone compn. having an absorbency of .ltoreq.1.0 at 270 nm and/or that of .ltoreq.0.5 at 280 nm measured in a 10 mm cell is prepd. by cyclocondensation reaction of a mixt. of NH<sub>3</sub>, monomethylamine, dimethylamine, and trimethylamine with a mixt. of .gamma.-butyrolactone at the methylamine content of .gtoreq.85% in the above amine mixt. This process satisfies more than one of the following 4 reaction conditions selected from (a) the difference between the metal compns. inside a distn. column and a distn. column packing material and those of the surfaces of the distn. column and distn. column packing material contacting the reaction mixt. introduced to the distn. purifn. system from the reaction process is maintained at .ltoreq.6%; (b) the .gamma.-butyrolactone mixt. does not possess the UV absorption higher than that of pure .gamma.-butyrolactone; (c) a no. of compds. other than .gamma.-butyrolactone possessing the concn. of .gtoreq.10 ppm measured by gas chromatog. using a polyethylene glycol-chem. bonded fused silica column are .ltoreq.20; and (d) a no. of compds. other than .gamma.-butyrolactone possessing the concn. of .gtoreq.10 ppm measured by gas chromatog. using a bis(cyanopropyl)methylsilane-chem. bonded fused silica column are .ltoreq.15. This process gives a N-methyl-2-pyrrolidone compn. having good hue, does not strongly discolor when used as solvent, and is useful as industrial solvent such as metal cleaning agent and solvent for functional polymers. Thus, 2,500 g .gamma.-butyrolactone and 2,956 g 32 wt.% aq. monomethylamine were added to an 10 L SUS autoclave and allowed to react at 240.degree. for 5 h to give a reaction mixt. contg. N-methyl-2-pyrrolidone 52, H<sub>2</sub>O 47, monomethylamine 1%, and other small amt. of byproducts which was continuously fed to a 15-stage Oldershaw column and distd. at 100.degree. (tower top temp.) and 120.degree. (the bottoms temp.) under normal pressure to give aq. monomethylamine soln. from the top of the column and an aq. N-methyl-2-pyrrolidone soln. from the bottom of the tower. The N-methyl-2-pyrrolidone soln. was redistd. using a 50-stage Oldershaw column to obtain water at a reflux ratio of 1 and pressure of 200 mmHg and N-methyl-2-pyrrolidone at reflux ratio of 1, pressure 20 mmHg, and 98.degree. (tower top temp.). The purified N-methyl-2-pyrrolidone had

99.97% purity and exhibited a UV absorbency of 0.116 at 270 and 280 nm in a 10 mm cell.

IC ICM C07D207-267

DT Patent

LA Japanese